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Multichannel Infrared (IR) Absorption Spectroscopy Applied to Solid Propellant Flames

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13. ABSTRACT (Maximum 200 words) A multichannel infrared (IR) absorption technique has been developed and applied to the study of solid propellant flames. Infrared absorption spectra of HCN, H ₂ O, N ₂ O, CO, CO ₂ , and CH ₄ have been obtained through the dark zone of solid propellant flames during steady-state combustion conditions through the use of a 1,024-element platinum silicide (PtSi) array detector. The experiment consists of a quartz tungsten halogen lamp as the source, a 0.320-m spectrometer with a 75-groove/mm grating, the PtSi array detector, and a windowed strand burner where cylindrical propellant samples are burned cigarette fashion in the presence of nitrogen buffer gas. Experimental results for the nitramine propellants XM39 and M43 and a double-base propellant JA2 are presented, and estimates for the temperature and species concentration are given through the use of the HITRAN database and associated PC programs. Comparisons are made between this work, other published measurements, and thermochemical equilibrium calculations.				
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1. INTRODUCTION

Although the steady-state combustion of solid propellants has been under investigation for a number of years, the detailed chemical and physical processes are still not well understood. Solid propellants burning at moderate pressures exhibit distinctly different visual characteristics depending upon their composition. Many propellants burn as a two-stage flame with a luminous portion and a nonluminous "dark zone" that is visually transparent (Kubota 1980). The length of this dark zone increases with decreasing pressure, and for sufficiently low pressures the luminous flame is not formed. Common gun propellants (i.e., single base [nitrocellulose], double base [nitrocellulose-nitroglycerin], and low-vulnerability nitramine propellants) exhibit dark zones which arise from the slow conversion of NO to N₂. The delayed ignitions which are found to take place in some gun firings are thought to be related to the chemistry occurring in the dark zone.

In order to understand the phenomena such as delayed ignition, as well as develop models for propellant combustion in general, it is necessary to measure the combustion processes occurring in the dark zone. Ideally, the technique which is employed to probe the dark zone should have the following features: (1) It should be performed under steady-state combustion conditions and *in situ*; (2) It should be nonintrusive; (3) The data acquisition time should be fast enough (<0.1 s) to minimize the fluctuations introduced by the unsteadiness of the propellant flame; (4) It should simultaneously provide temperatures and absolute concentrations of the chemical species present; (5) The spatial resolution should be fine enough so that the dark zone may be profiled in detail; (6) The technique should have sufficient sensitivity to detect species present in small quantities ($\approx 1\%$).

Over the past few years, this laboratory has developed a multichannel optical absorption technique which incorporates many of the previously listed features. Results for NO and OH have been obtained for double base and nitramine propellants using a multichannel array detector sensitive in the ultraviolet-visible (UV-VIS) spectral region (Vanderhoff 1991; Vanderhoff, Teague, and Kotlar 1992b). Recent advances in multichannel detectors allow us to extend this optical absorption technique into the mid-infrared (IR) spectral region. Since many of the molecules produced during the combustion of solid propellants do not have ultraviolet (UV) or visible transitions that can be easily monitored, it is advantageous to probe the IR portion of the spectral region. In this report we demonstrate viability of the multichannel IR absorption technique as applied to the combustion of solid propellant flames.

Consequently, we report here absorption spectra for XM39, M43, and JA2 and estimate the temperature and concentrations for HCN, H₂O, CH₄, N₂O, and CO.

2. EXPERIMENTAL

The absorption experiment and the windowed pressure vessel used in this study have been described in detail previously (Vanderhoff 1991; Vanderhoff, Teague, and Kotlar 1992b), and so only pertinent changes to the experiment are described here. The schematic diagram representing the experimental setup is given in Figure 1. A 0.320-m Czerny-Turner-type spectrometer (J-Y Systems, model HR-320) has been employed and equipped with a 75-groove/mm grating blazed at 4 μm . The liquid nitrogen-cooled array detector (Princeton Instruments) is composed of 1,024 platinum-silicide (PtSi) elements in a linear configuration. Each element is $15 \times 2,500 \mu\text{m}$ in size and spaced on 25- μm centers. When operated in first order, the system has a total spectral window of approximately 1.0 μm and a spectral resolution of 0.007 μm using a 125- μm entrance slit width. The spectral calibration was performed using a low pressure argon (Ar) lamp. The quantum efficiency for PtSi as reported by the manufacturer is quite low (Cizdziel 1991). The detector is sensitive from about 0.9 to 4.5 μm with a maximum quantum efficiency (QE) of 8% at 1.2 μm and decreases to about 2% at 2.5 μm . At wavelengths beyond 3 μm the QE drops to 1% or less. Although the low QE is somewhat of a drawback, PtSi arrays exhibit extremely good pixel-to-pixel uniformity, allowing them to perform comparably to other types of IR detectors (Mooney and Dereniak 1987). When used with the Princeton Instruments ST-120 controller, a scan rate of 10 μs /diode (i.e., 10.24 ms needed to readout entire diode array) is obtained with a dynamic range of 14 bits.

Light produced from a 250-W quartz tungsten halogen lamp (Osram HLX 64655) was used as the source and was chosen for its stability as well as for its small filament size ($7.0 \times 3.5 \text{ mm}$). Although the quartz lamp bulb cuts off direct radiation from the filament at wavelengths longer than 3.5 μm , the hot envelope acts as a broad band emitter with output further into the IR. CaF₂ lenses were used to direct the light into the chamber, and then collect and focus it onto the entrance slit of the spectrometer. An IR long-pass filter was used between the sample chamber and the spectrometer to remove any higher order light, as well as any visible emission emanating from the burning propellant. A fast shutter, with a minimum opening time of 1.8 ms (Vincent Associates Model LS6), was placed between the light source and the chamber so that a background spectrum, consisting of the background, the dark charge, and any emission from the sample, could be subtracted from the spectrum of the transmitted light. Radiation

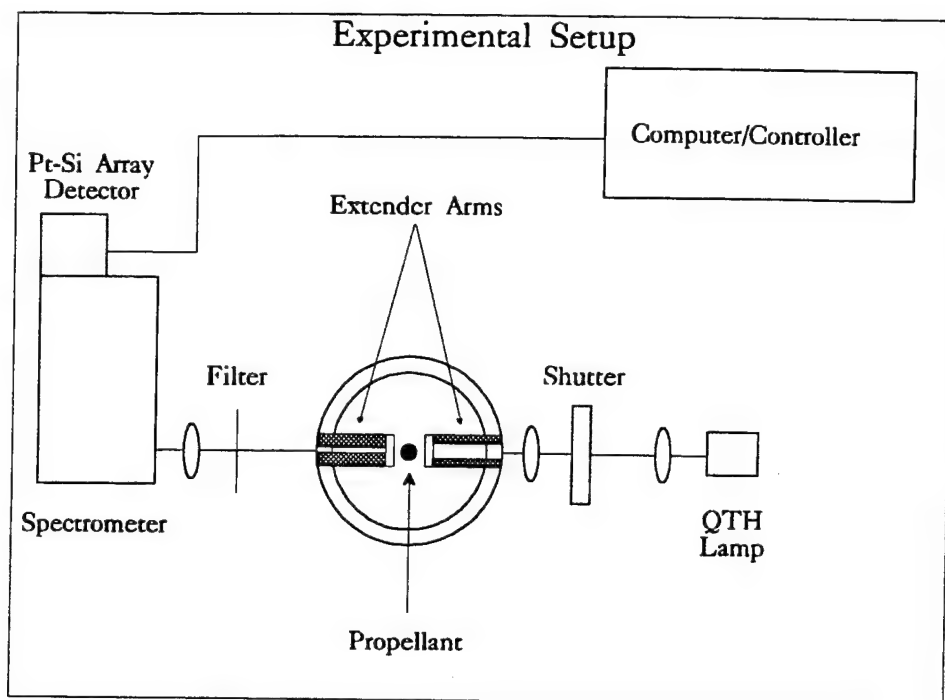


Figure 1. Schematic diagram of the experimental setup used in obtaining the infrared absorption spectra of solid propellant flames.

emitted from the flame is more of a problem in the IR region than it was in the UV-VIS region, and so the ability to subtract it from the transmitted light is essential for obtaining quantitative transmission data. The sample is ignited through a ZnSe optical window at the top of the chamber using a 25-W CO₂ laser (Synrad Inc.). Although the space surrounding the propellant sample is purged with flowing nitrogen (Vanderhoff 1991), some combustion products still migrate to this region. These products have significantly lower temperatures (near room temperature) and their contributions to the absorption spectra cause significant reductions in the observed temperature of the system. In order to minimize the recirculation of the combustion products into the light path outside of the combustion region, the overall path length was reduced to a length approximately 0.4 cm longer than the propellant sample diameter and was accomplished with the use of extender arms with sapphire windows mounted inside the chamber (see Figure 1). When possible, propellant samples with a larger diameter were used (1.08 cm vs. 0.65 cm) to increase the ratio of the sample length to total path length, which also helps to minimize the contribution of any cold products which have migrated outside the combustion region. The overall spatial resolution of 0.250 cm was set by the inside diameter of the extender arm on the spectrometer side. All spectra obtained in this study were acquired at an average height 0.125 cm directly above the propellant surface.

A four-channel digital delay generator serves as the master clock in the experiment, synchronizing the ST-120 controller with the operation of the shutter and the triggering of the propellant feed mechanism. The general timing diagram is shown in Figure 2. When operated in the external synchronized mode, the ST-120 controller will read and reset every diode of the array upon the receipt of the falling edge of the external synchronization pulse. In the present experiment, the frequency of the master clock is set to 95 Hz, so that the array is read and reset every 10.53 ms. This means that a complete spectrum is collected every 10.53 ms. As mentioned previously, a shutter allows a background spectrum to be acquired to minimize the contamination of the transmitted light signal by the emission of the propellant flame. When the shutter is open, a spectrum which measures the transmitted intensity of light (I_s) is collected. When the shutter is closed, a spectrum of the flame emission and background (B_s), which can then be subtracted from I_s for background correction during postprocessing of the spectra, is acquired. The emission spectrum is calculated by subtracting the background collected when the propellant is not burning (B_o) from the background collected while the propellant is burning (B_s). In order to compensate for the finite time required by the shutter to open or close, the frequency of the shutter is set to one-fourth of the frequency of the master clock. This allows every other spectrum to be acquired while the shutter is either fully opened or fully closed. The time required to acquire one data pair consisting of one signal spectrum, I_s , and one background spectrum, B_s , is 42 ms when signal averaging is required or 32 ms for a single shot experiment. The ability to acquire both IR absorption and emission spectra sequentially at a fast acquisition rate and with low noise provides a significant advantage over other diagnostic techniques which are currently being used to study solid propellant flames.

A typical experimental run involves the following sequence of events. The propellant sample is positioned inside the chamber so that the transmitted light (I_o) and the background (B_o) can be collected after the chamber is sealed and pressurized. The sample is then moved into position so that the top of the propellant just blocks the light beam from passing through the chamber. The CO_2 laser is pulsed for approximately 0.1 s, thereby igniting the propellant. The master clock is triggered, which, in turn, starts the propellant feed mechanism, the data acquisition process, and the shutter cycle. The ST-120 controller will then collect a predetermined number of spectra, which include the spectra when the shutter is either fully open or closed as well as the spectra where it is only partially opened. The errant spectra where the shutter is not fully opened (or closed) are then deleted during the postprocessing routine. The total data accumulation time can be varied depending upon the length of the propellant samples used, the rate at which they burn, and the amount of averaging needed to obtain a good signal-to-noise (S/N) ratio.

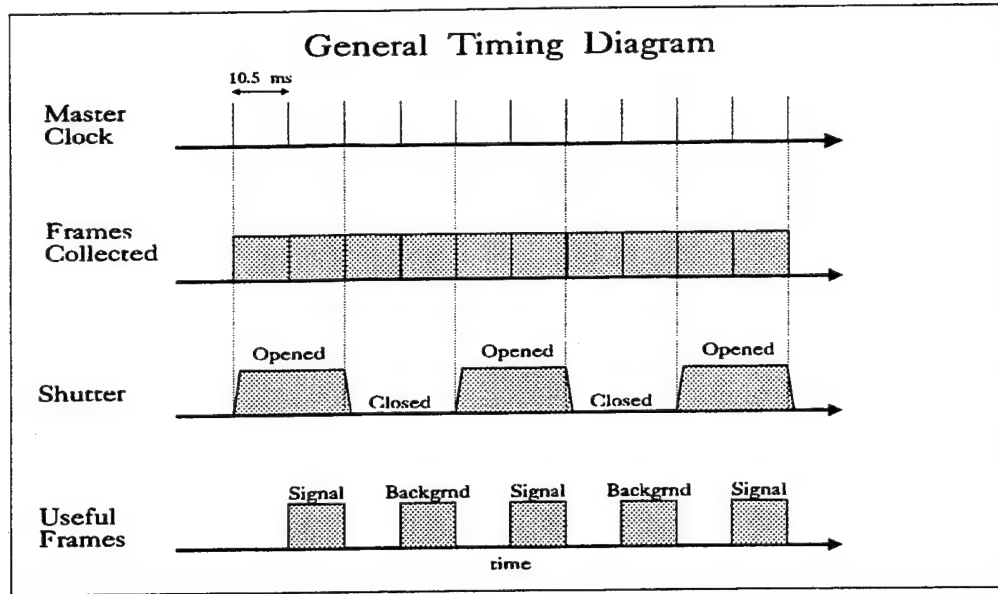


Figure 2. General timing diagram used for data acquisition in the infrared absorption experiment.

Although under certain conditions the S/N ratio was high enough that no averaging was required (i.e., single shot spectra), the spectra presented here are the average of 100 spectra and took approximately 4 s to acquire. The final transmission spectrum, T_{av} , was calculated via

$$T_{av} = \frac{\sum_i^n \left[(I_s - B_s) / (I_o - B_o) \right]_i}{n}$$

The emission spectra are processed by taking the average of difference between the background spectrum taken during combustion, B_s , and the background spectrum taken before combustion, B_o ,

$$E_{av} = \frac{\sum_i^n \left[(B_s) - (B_o) \right]_i}{n}$$

As was found in the UV-VIS absorption investigations (although not nearly as large), a broadband attenuation (absorption) of the transmitted light beam was observed in the IR spectra. The source of the attenuation is assumed to be scattering from particulates and/or broadband absorption of large molecules (i.e., soot). A more complete description of the background correction can be found elsewhere (Vanderhoff, Teague, and Kotlar 1992b). As before, the broadband attenuation will be treated as a function of the pathlength and only weakly dependent upon wavelength so that the absorption can be approximated as

$$I(w) = I_0 \exp[-k_w l - b_w l] \approx I_0 B(w) \exp[k_w l],$$

where $I(w)$ is the transmitted intensity of light, I_0 is the incident light intensity of the lamp, k_w is the absorption coefficient for the molecule of interest, l is the pathlength for absorption, and $B(w)$ is the baseline correction factor. The observed shape of the experimental baseline is used to determine the appropriate form of $B(w)$. The experimental spectra are then multiplied by the baseline correction factor to obtain the background corrected spectra used for the estimation of the concentrations and temperatures.

3. RESULTS AND DISCUSSION

The transmission spectra of XM39 propellant taken in the dark zone region approximately 1.25 mm from the propellant surface during steady-state combustion conditions at 1.01 MPa nitrogen are shown in Figure 3. Five species (HCN, H₂O, CH₄, CO, CO₂, and N₂O) have been identified as exhibiting absorption over this wavelength region. Absorption bands of water are found in the 4,000–3,200-cm⁻¹ and 4,800–5,600-cm⁻¹ regions and can be assigned to the fundamental vibrational bands of ν_1 (3,657 cm⁻¹) and ν_3 (3,756 cm⁻¹) and combination band, $\nu_2 + \nu_3$ (5,332 cm⁻¹). Other readily identifiable bands include the fundamental CH asymmetric stretch (3,311 cm⁻¹) of HCN; the fundamental CH stretch (3,020 cm⁻¹) of CH₄; the fundamental of CO₂, ν_3 (2,349 cm⁻¹); the first overtone, $2\nu_1$ (2,563 cm⁻¹), of N₂O; and the first overtone, $2\nu_1$ (4,292 cm⁻¹), of CO. Line structure in the region of HCN can be attributed to that of H₂O. Evidence supporting this statement comes from the fact that the spacing of these lines matches that of the other water lines which are not overlapped with the HCN band. An estimate of the temperature can be obtained from a band spread analysis of the P and R branches of HCN and the Q and R branches of CH₄, which results in a temperature of 1,150 K \pm 50 K (Chan 1974). This value is consistent with

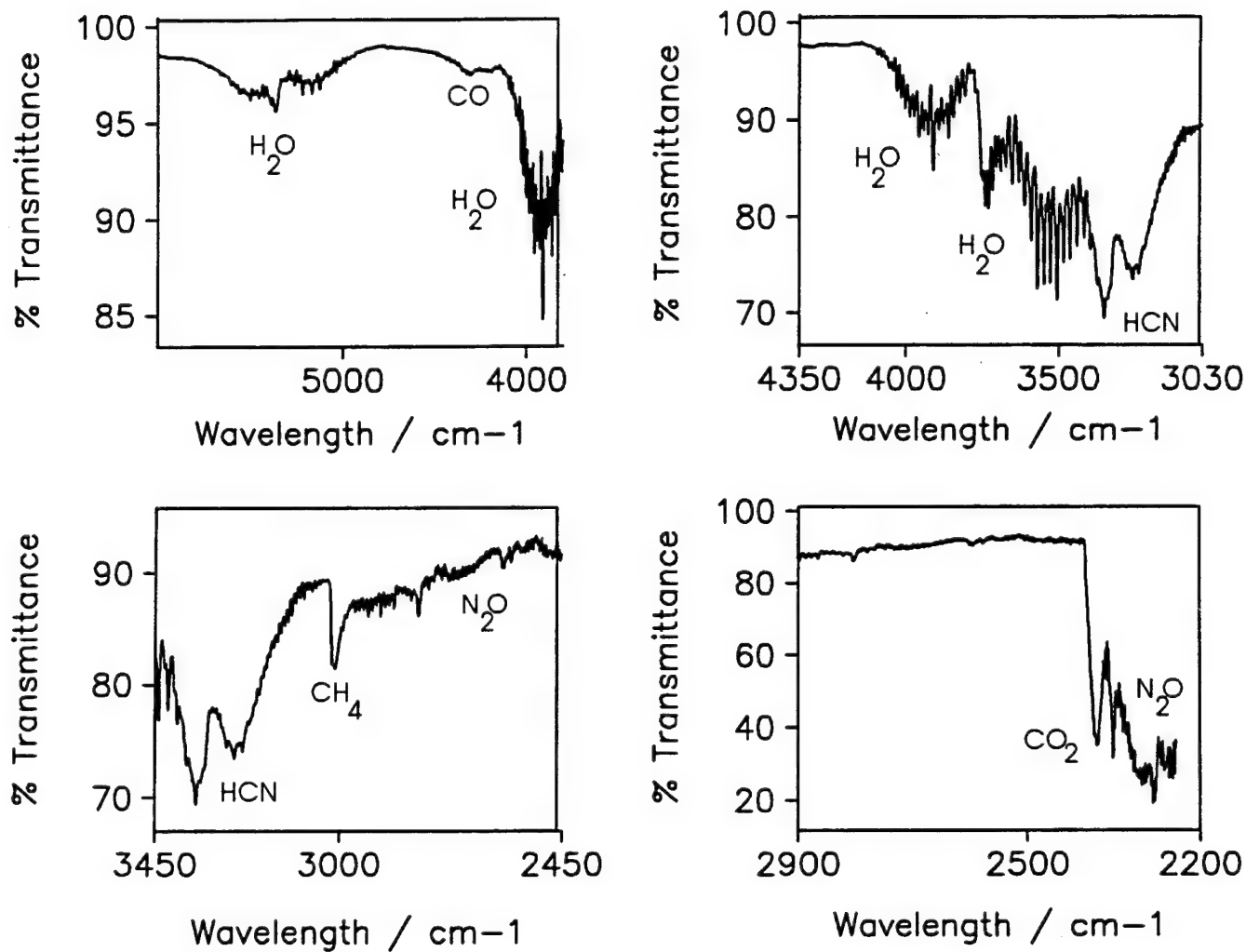


Figure 3. Transmission spectra of XM39 burning in 1.01 MPa nitrogen taken in the dark zone region at approximately 0.125 cm above the propellant surface.

previous dark zone temperature measurements for XM39 burning in 1.01 MPa of nitrogen (Teague, Singh, and Vanderhoff 1993).

Figure 4 shows an uncorrected emission spectrum of XM39 burning in 1.01 MPa extracted from the background frames collected during the acquisition of the transmission spectra presented in Figure 3. The spectrum displays the same spectral features as in the transmittance spectrum. A band spread analysis of the HCN emission band shows a temperature of 1,150 K in agreement with the transmission spectrum. As can be seen in Figure 4, a nonnegligible amount of emission from the flame reaches the detector during data acquisition. If this emission is not taken into account, significant distortions in the transmittance spectrum can result. An example of this is shown in Figure 5, which compares the transmission spectrum of XM39 with and without the correction for the emission. For this region, errors as large as 8% in the transmittance can be obtained if the emission is not corrected. This illustrates dramatically the advantages of our technique of collecting a background frame for each frame of transmitted light. Although we have not done so here, it is possible to extract a temperature for the system through the comparison of the calculated normalized radiance (obtained from the ratio of the transmission and corrected emission spectra) with that of a theoretical blackbody function (Solomon et al. 1986). This could serve as another check for the estimation of the temperature. Under the current experimental conditions, the background due to the soot was not a major problem. However, at lower pressures there is a substantial increase in amount of soot produced for both the nitramine and double base propellants. When a transmission and emission spectrum can be obtained with a substantial amount of soot particles, the temperature and concentration of the soot can be estimated as well.

The transmission spectra of M43 propellant taken in the dark zone region during steady-state combustion conditions where the surrounding environment is nitrogen gas maintained at a pressure of 1.01 MPa are shown in Figure 6. M43 is very similar to XM39 in composition, and the transmission spectra are also similar, as expected.

The transmission spectra of the double-base JA2 propellant also taken in the dark zone during steady-state combustion conditions at 1.01 MPa nitrogen are shown in Figure 7. As with the nitramine propellants, the water, CO, and CH₄ bands are readily identified. At these low pressures, JA2 burns at a much faster rate than the nitramine propellants, hence the S/N ratio is much worse in the JA2 spectra especially in the lower energy region. The most notable difference between the spectra of JA2 and the nitramines is the absence of the HCN band at 3,300 cm⁻¹. The lack of HCN in the spectrum for JA2 is

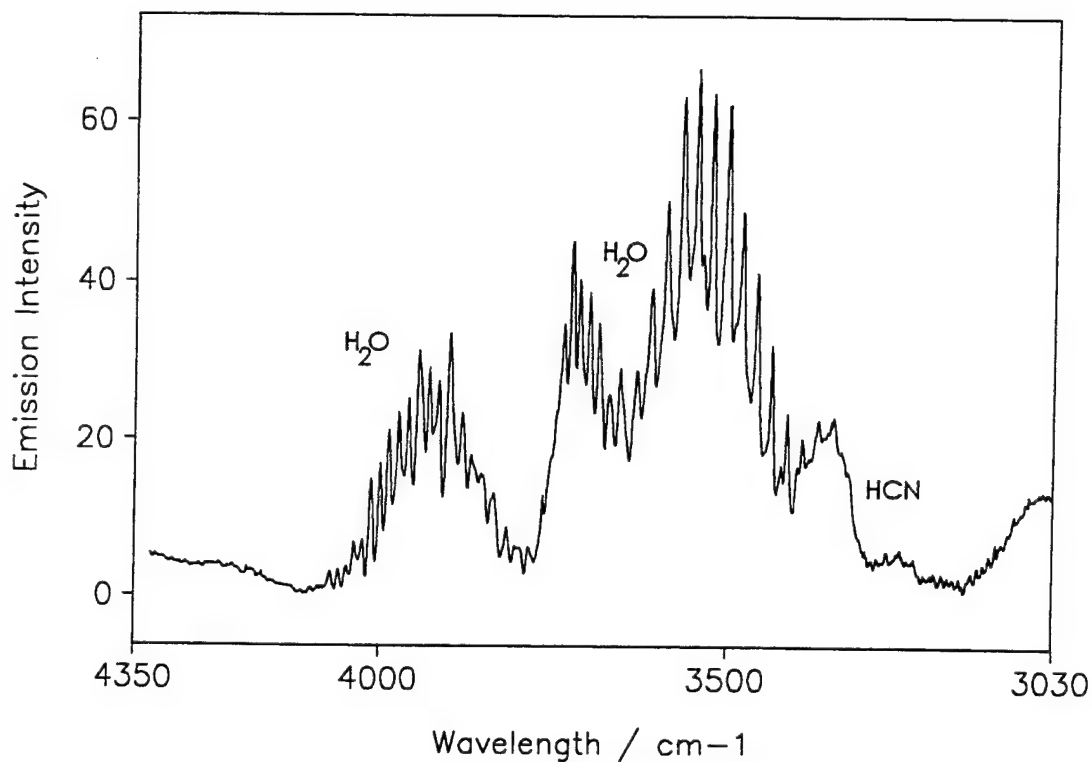


Figure 4. Uncorrected emission spectrum of XM39 burning in 1.01 MPa nitrogen extracted from the background frames collected during the acquisition of the transmission spectra in Figure 3.

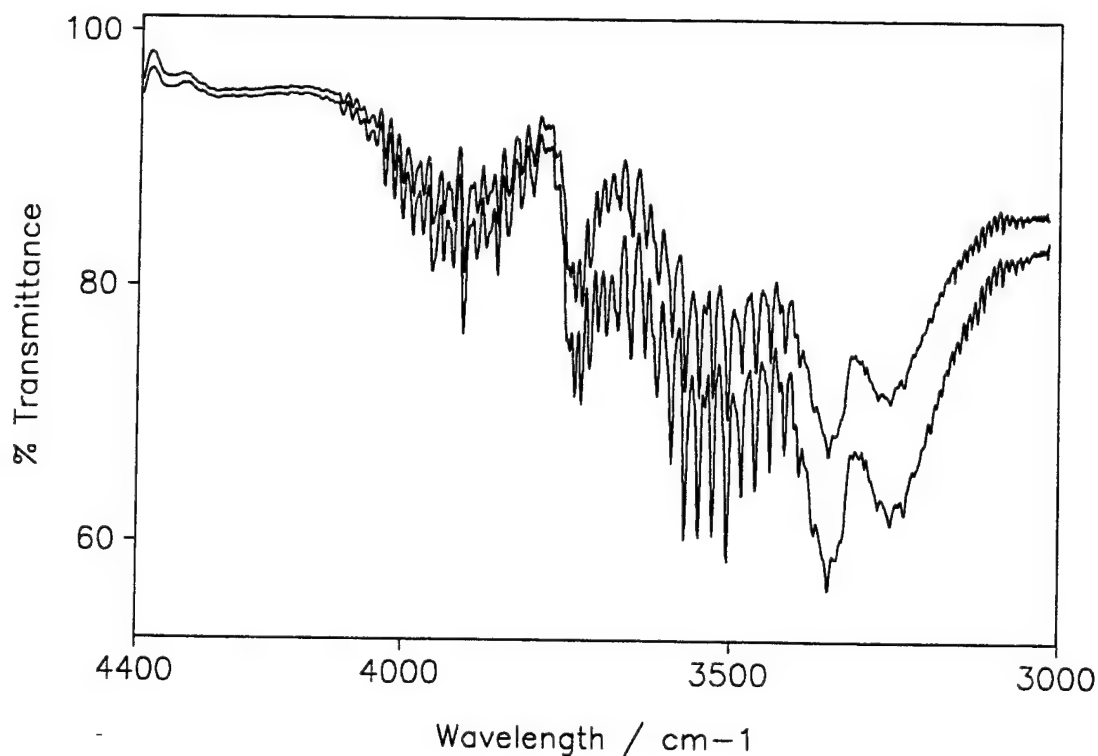


Figure 5. Comparison of the transmission spectrum of XM39 with (bottom) and without (top) the subtraction of the emission.

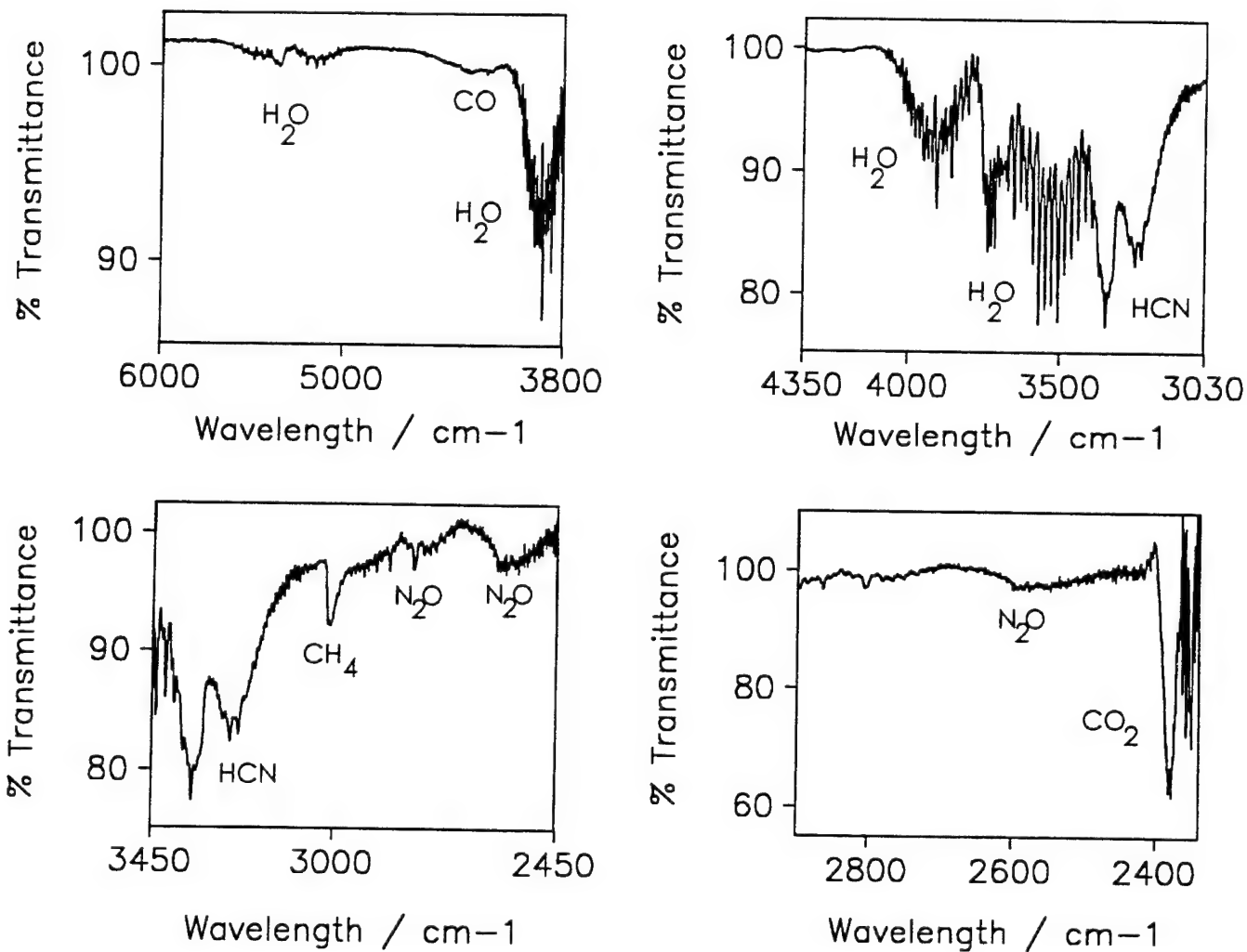


Figure 6. Transmission spectra of M43 burning in 1.01 MPa nitrogen taken in the dark zone region at approximately 0.125 cm above the propellant surface.

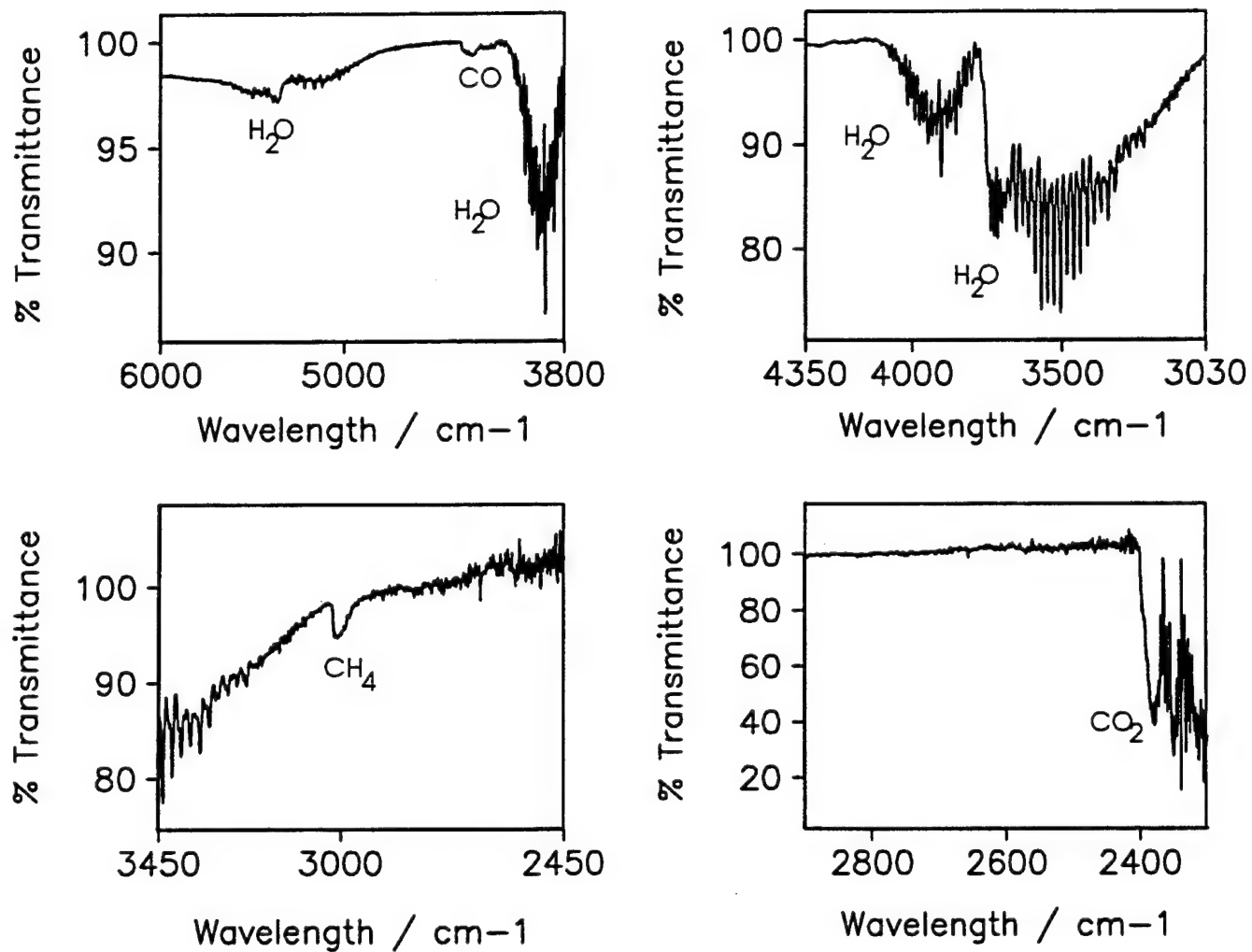


Figure 7. Transmission spectra of JA2 in 1.01 MPa nitrogen taken in the dark zone region at 0.125 cm above the propellant surface.

not surprising since there are no C-N bonds in its major components, nitroglycerine, nitrocellulose, and DEGDN. N_2O bands were also absent from the JA2 spectra.

Although not shown here, the nitramine propellant HMX2 and the double-base propellant M9 have also been surveyed, and results show that the spectra for these propellants are qualitatively similar to the spectra shown here for XM39 and JA2, respectively.

The HITRAN database with associated PC programs (Rothman et al. 1992) was used to synthetically produce a spectrum for M43 over the spectral region of $4,350\text{--}3,030\text{ cm}^{-1}$ similar to that found in Figure 5 and is presented in Figure 8. The input parameters used are the total pressure (1.01 MPa), the estimated temperature (1,150 K), the path length (0.65 cm), and the spectral resolution ($0.007\text{ }\mu\text{m}$). An instrument slit response function was generated from an Ar emission line in the spectral region of interest using a low pressure Ar lamp and convoluted over the spectrum generated by HITRAN, producing the synthetic spectrum shown in Figure 8b, along with the spectrum from the experiment for comparison (Figure 8a). The concentrations, in mole-percent,* of HCN, H_2O , and CH_4 were adjusted to best simulate the experimental spectrum over the same region. The concentrations of CO and N_2O were estimated in a similar manner, and all of the results are identical to those obtained for XM39. An estimation could not be made for CO_2 due to the saturation of this band. The uncertainty of these estimates for the concentrations vary with species but are, in general, expected to be reliable to 30%. Because an estimation of the temperature for JA2 could not be calculated using the band spread method for HCN, the temperature was estimated from previous UV-VIS absorption experimental measurements of NO^3 .

It has been suggested that there should be a temperature gradient over the absorption path length within the chamber. This temperature gradient is probably due to (1) the cooling of the propellant about the edges by the flowing shroud gas, and (2) the migration of cold combustion gases back into the absorption path volume between the propellant and the windows. Evidence for the cooling of the propellant by the shroud gas is demonstrated by the visual observation of the cupping of the surface of the propellant as it burns. Because the multichannel absorption technique is a line-of-sight measurement, the light is integrated along a line passing through the center of the propellant, producing a spectrum that is a composite which reflects the temperature and species variations along this line. In order to better

* The experiment is carried out under constant total pressure conditions; thus, mole-percent represents the relative amount of each species. Total pressure and mole-percent of species present give absolute concentration.

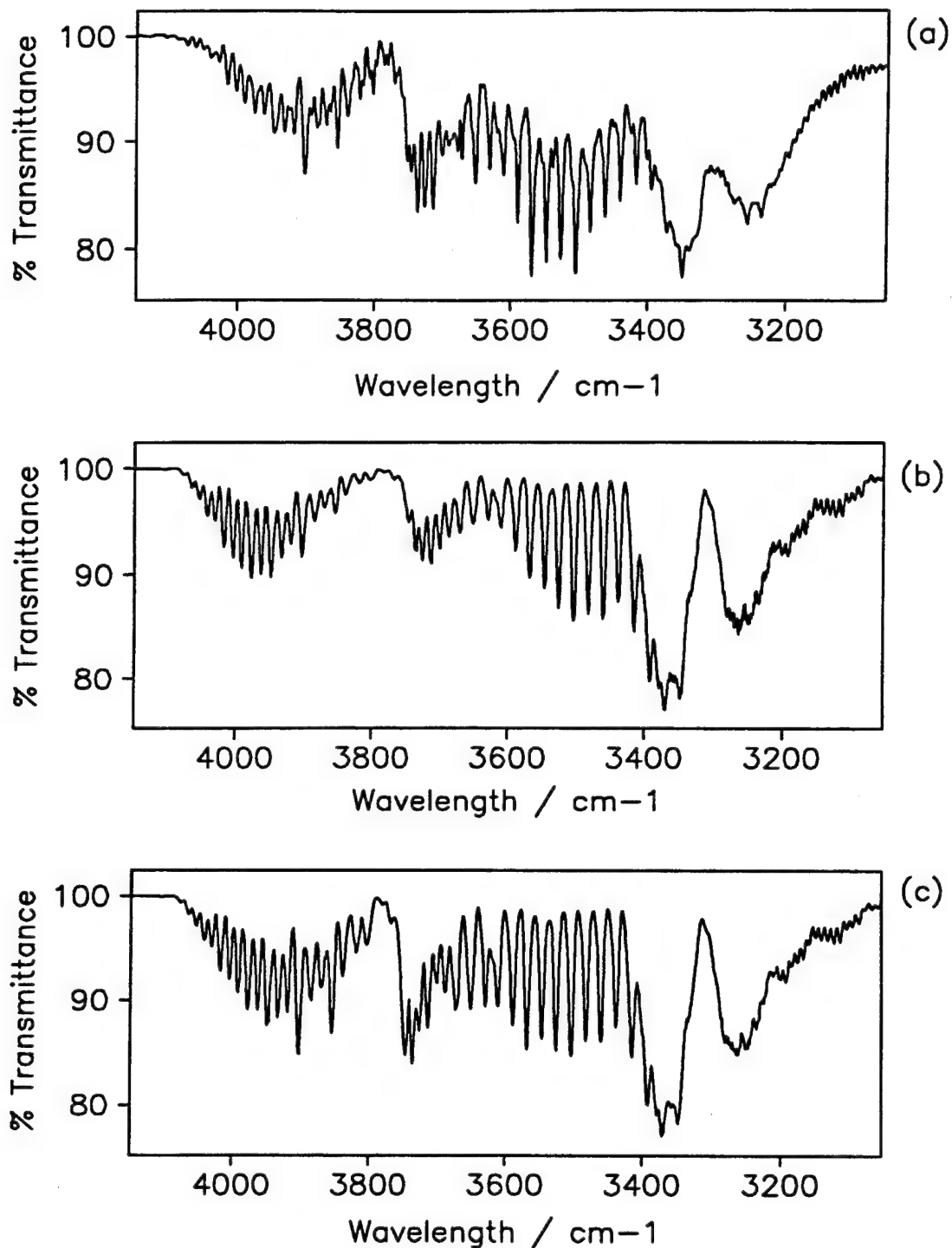


Figure 8. Comparison of (a) the transmission spectrum of M43 burning in 1.01 MPa nitrogen taken in the dark zone region 0.12 cm above the propellant surface with the synthetic spectra (b) and (c) produced with the HITRAN database. (The parameters used for the synthetic transmission spectra are as follows: (b) P = 1.01 MPa, T = 1,150 K, with mole-percentages of HCN: 18%; H₂O: 5%; CH₄: 1.5% and (c) P = 1.01 MPa, T = 1,150 K, with mole-percentages of HCN: 18%; H₂O: 5%; CH₄: 1.5%; plus H₂O: 2.5% at 400 K. See text for details.)

understand this aspect of the measurement, we have used the HITRAN program to simulate spectra with two temperatures to see if a better representation of the experimental spectrum can be produced. One of these simulations is shown in Figure 8c. The spectrum is a composite of a spectrum produced with a temperature of 1,150 K and mole fractions of HCN: 18%; CH₄: 1.5%; and H₂O: 5% added to another spectrum produced with a temperature of 400 K and a mole fraction for H₂O of 2.5%. As can be seen in the figure, the R branch of the ν_3 band of H₂O (3,800–4,100 cm⁻¹) is simulated much better with a two-temperature model for water than with a one-temperature model. However, a two-temperature model for HCN and CH₄ with the same proportional concentrations for the two temperatures as found for water (i.e., 2 [1,150 K]:1 [400 K]) proves to be much worse in simulating the experimental spectrum. We are currently developing a multitemperature nonlinear least-squares fitting routine along with the incorporation of additional hot bands and higher J values not yet included in the HITRAN database. These modifications should improve the certainty for the estimates for the temperatures and concentrations reported here.

4. COMPARISONS

To place the present data in context with other published results, Tables 1 and 2 have been developed. These two tables contain relevant experimental parameters, experimental and estimated dark zone data and calculated thermochemical equilibrium temperatures. Unfortunately, there is not an abundance of experimental data from which to select, thus the comparisons may not be strictly valid. Before making any detailed comparisons, general differences in the data sets are given. Table 1 contains information on nitramine composite propellants; five entries are RDX-based propellants, and two are HMX-based. Table 2 contains information for homogeneous double-base propellants. The data obtained for pressures of 9 atm and above are taken during steady-state combustion conditions whereas the 1-atm pressure data are taken with laser assisted combustion conditions. The data sets are obtained from a variety of analytical techniques: mass spectrometry with micro probe sampling, grab sampling with post analysis, and absorption spectroscopy under steady-state combustion conditions. Each of these techniques has strong and weak points. Although mass spectrometric sampling provides data on most species; the probe is intrusive, multiple species appear at the same charge-to-mass ratio, and the ionization process can fragment the species, thus complicating the analysis. Grab sampling with post analysis is intrusive and without special heating measures can miss condensable constituents such as H₂O and CH₂O. Absorption spectroscopy is a line-of-sight technique and suffers from overlapping spectra, especially in the IR regions.

Table 1. Comparison of Experimentally Determined Dark Zone Temperatures and Species Concentrations for Nitramine-Based Solid Propellants (Adiabatic Equilibrium Flame Temperatures Are Shown in the Last Two Rows; Values in Parentheses Are Estimates [Guesses])

Parameters	XM39 ^a	XM39 ^b	XM39 ^c	M43 ^c	M43 ^b	HMX2 ^d	HMX-PE ^e
P(atm)	10	21.7	1	1	9.8	19	20
Ht(cm)	0.15	0.05	0.5	0.5	0.05	0.05	dark zone
T _{DZ} (K)	1150	947	1170	1240	1234	1300	1300
NO	0.16	0.08	0.22	0.23	0.06	0.10	0.17
CO	≤0.15(.15)	0.08	0.08	0.09	0.16	—	0.15
H ₂	(.05)	(.05)	0.015	0.02	(.05)	—	0.05
N ₂	(.315) ^f	(.44) ^f	0.08	0.08	(.30) ^f	—	0.08(.13)
H ₂ O	0.05	0.07	0.21	0.21	0.14	—	0.20 ^g
CO ₂	(.05)	0.04	0.05	0.04	0.05	—	0.06
NO ₂	(0)	(0)	0.0	0.0	(0)	—	(0)
N ₂ O	<0.07(.03)	(.03)	0.03	0.04	(.03)	—	(.03)
HCN	0.18	(.20)	0.22	0.22	(.20)	—	(.20)
CH ₄	0.015	(.01)	0.01	0.01	(.01)	—	(.01)
CH ₂ O	(0)	(0)	0.06	0.06	(0)	—	(0)
T _{NL-S} (K)	2161	2163	2151	2416	2449	2080	1928
T _{NL-DZ} (K)	2518	1755	2723	2808	1864	—	2711

^a Present data; also Teague, Singh, and Vanderhoff (1993).

^b Data of Mallory and Thynell (1994).

^c Data of Kuo (1994) (ARO/URI Program Review) and Chin-Jen Tang and Litzinger (1994).

^d Data of Vanderhoff, Teague, and Kotlar (1992a).

^e Data of Kubota (1982).

^f Nitrogen added to bring the total mole fraction to 1.0; otherwise the thermochemical equilibrium program will renormalize.

^g A 20% H₂O concentration is assumed.

Table 2. Comparison of Experimentally Determined Dark Zone Temperatures and Species Concentrations (Mole Fraction) for Double-Base Solid Propellants (Adiabatic Equilibrium Flame Temperatures Given in the Last Two Rows)

Parameters	JA2 ^a	JA2 ^b	M9 ^b	M9 ^c	DB1 ^d	DB2 ^e
P (atm)	10, 16	1	1	17	14.6	9
Ht (cm)	0.15	0.2	0.2	0.1	0.6	0.2
T _{DZ} (K)	1450	1890	1900	1500	1575	1500
NO	0.24	0.31	0.33	0.31	0.24	0.21
CO	0.26	<0.36(.32)	<0.33(.32)	—	0.30	0.38
H ₂	(.08)	0.04	0.06	—	0.08	0.08
N ₂	(.33) ^f	(.02)	(.02)	—	0.065	0.02
H ₂ O	0.08	0.14	0.16	—	0.20 ^g	0.20 ^g
CO ₂	--(.1)	0.10	0.10	—	0.10	0.09
NO ₂	(0)	0.015	<0.005	—	(0)	(0)
CH ₄	0.01	(.01)	(.01)	—	0.07	0.026
C ₂ H ₄	(0)	(0)	(0)	—	0.08	0.008
CH ₂ O	(0)	0.045	0.015	—	(0)	(0)
T _{NL-S} (K)	2793 at 16 atm	2678	2800	3023	3075	2813
T _{NL-DZ} (K)	2947 at 16 atm	2990	2978	—	3057	2859

^a Present data; also Vanderhoff et al. (1992a).

^b Data of Liiva, Fetherolf, and Litzinger (1991).

^c Data of Vanderhoff, Teague, and Kotlar (1992a).

^d Data of Heller and Gordon (1955).

^e Data of Lengelle et al. (1984).

^f Nitrogen added to bring the total mole fraction to 1.0; otherwise the thermochemical equilibrium program will renormalize.

^g A 20% H₂O concentration is assumed.

Having mentioned some of the weaknesses in the data sets, a more detailed look is now presented. The homogeneous double-base propellant data of Table 2 are in better shape than the data of Table 1. That is, there is better agreement among data sets and energy conservation conditions. The last two rows (T_{NL-S}[K], T_{NL-DZ}[K]) are a measure of the energy content. The NASA-Lewis thermochemical equilibrium code (Svehla and McBride 1973) is used to calculate the adiabatic flame temperatures from two starting points: (1) the solid propellant ingredients (see Appendix) and is designated T_{NL-S}(K) and (2) the

experimental dark zone species concentration and temperature measurements designated $T_{NL-DZ}(K)$. Where the measurements are incomplete, estimates (guesses) are made for these concentrations and enclosed in parentheses. Ideally, $T_{NL-S}(K)$ and $T_{NL-DZ}(K)$ should be the same. Differences reveal uncertainties in the experimental dark zone data or equilibrium assumptions. The data in columns L and M of Table 2 demonstrate excellent energy matching. Columns H, I, and J show substantial differences in these calculated equilibrium temperatures, but the experimentally measured dark zone temperatures in columns I and J are well above the results in the other columns. We feel that the dark zone temperature values should be several hundred degrees lower (consistent with columns H, K, L, and M), which would then put the equilibrium temperatures in better agreement. Column H represents present and previous data obtained in this laboratory. These data result in about a 150° difference in the equilibrium temperatures. Not all of the concentrations are accounted for in these measurements, as can be seen by the need to add nitrogen to make a total mole fraction of one. There is disagreement in the H_2O concentration measurements between columns H and I; also with respect to the nitramine propellants of Table 1. No explanation for the variation in H_2O concentration is known. There could be experimental difficulties in proper measurements for condensable species. Pressure effects might be present, but thermochemical equilibrium calculations do not support this possibility. Formaldehyde shows up in the low pressure data but is absent at higher pressure, which is consistent with expectations.

Nitramine propellant data are shown in Table 1, and here most of the columns give $T_{NL-DZ}(K)$ calculated values much in excess of those calculated, starting with the solid propellant ingredients. In general, these propellants combust to give fuel-rich mixtures. Hence, energy (temperature) values are particularly sensitive to oxidizer content. For double-base propellants, there is only one major dark zone species that is not an equilibrium species—namely NO. In other words, experimental measurements show that NO is a major dark zone species whereas thermochemical equilibrium codes predict only trace amounts of NO. Nitramine propellants have two major nonequilibrium species: NO and HCN, which adds to the difficulty in obtaining appropriate experimental results.

Over the last few years, significant additions to the total amount of solid propellant combustion diagnostic data have occurred. Nonetheless, consistent measurements are still needed for the major species of nitramine propellants. It appears that there is reasonable agreement only with respect to HCN and CO_2 . In order to bring down $T_{NL-DZ}(K)$, a reduction in oxidizer content or lower dark zone temperatures or both are required. The temperature measurements are reasonably consistent so a lowering of the oxidizer content appears to be the more probable solution. Columns B and E have the lowest measured NO

concentrations, and here $T_{NL-DZ}(K)$ is substantially less than $T_{NL-s}(K)$. Column A contains our present measurements for XM39. Within experimental error M43 propellants give identical results. At present only maximum estimates are given for CO and N_2O concentrations. Improvements in the experiment as well as data analysis have progressed to the point that we foresee reporting actual values in the near future.

5. SUMMARY

A multichannel IR absorption technique has been developed and applied to study of solid propellant flames. Spectra have been reported for the nitramine propellants XM39 and M43 and the double-base propellant JA2. A temperature and concentrations for HCN, H_2O , CH_4 , N_2O and CO have been estimated using the HITRAN database. When compared to the literature data obtained with laser-assisted heating, the results were found to be qualitatively similar. Future work will involve a continued survey of additional propellants, improvements in the data analysis through the use of a nonlinear least squares technique, and an increase in the spatial resolution of the system in order to begin profiling the dark zone.

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APPENDIX:

**INGREDIENTS, HEATS OF FORMATION, AND HEATS OF EXPLOSION
FOR THE VARIOUS SOLID PROPELLANTS DISCUSSED**

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Table A-1. Ingredients, Heats of Formation, and Heats of Explosion for the Various Solid Propellants Discussed (The Nitration Level N Is Also Given for Nitrocellulose)

Propellant Type	Ingredients	Weight-Percent (cal/mol)	HOF	HOE (cal/g)
XM39	RDX ^a	76	14,690	830
	Cellulose Acetate Butyrate	12	-255,800	
	Nitrocellulose (12.6% N)	4	-169,160	
	Acetyl Triethyl Citrate	7.6	-415,000	
	Ethyl Centralite	0.4	-25,100	
M43	RDX	76	14,690	—
	Cellulose Acetate Butyrate	12	-255,800	
	Nitrocellulose (12.6% N)	4	-169,160	
	Energetic nitro plasticizer	8	—	
HMX2	HMX ^b	80	17,920	780
	PU Binder	20	-118,300	
HMX-PE	HMX	80	17,920	—
	PE Binder	20	-72,000	
JA-2	Nitrocellulose (13.04% N)	58.2	-165,580	1,120
	Nitroglycerin	15.8	-88,600	
	DEGDN ^c	25.2	-103,500	
	AKARDIT II	0.05	-25,500	
M9	Nitrocellulose (13.29% N)	57.6	-163,460	1,308
	Nitroglycerin	40.02	-88,600	
	Ethyl Centralite	0.73	-25,100	
	Potassium Nitrate	1.63	-117,760	
DB1	Nitrocellulose (12.6% N)	55	-169,600	1,320
	Nitroglycerin	45	-88,600	
DB2	Nitrocellulose (11.6% N)	52	-176,690	1,100
	Nitroglycerin	43	-88,600	
	Ethyl Centralite	3	-25,100	

^a RDX: cyclotrimethylene-trinitramine.

^b HMX: cyclotetramethylene-tetranitramine.

^c DEGDN: diethyleneglycoldinitrate.

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5. Has the information in this report led to any quantitative savings as far as man-hours or dollars saved, operating costs avoided, or efficiencies achieved, etc? If so, please elaborate. _____

6. General Comments. What do you think should be changed to improve future reports? (Indicate changes to organization, technical content, format, etc.) _____

CURRENT
ADDRESS

Organization

Name

Street or P.O. Box No.

City, State, Zip Code

7. If indicating a Change of Address or Address Correction, please provide the Current or Correct address above and the Old or Incorrect address below.

OLD
ADDRESS

Organization

Name

Street or P.O. Box No.

City, State, Zip Code

(Remove this sheet, fold as indicated, tape closed, and mail.)
(DO NOT STAPLE)